

# Optical Constants and Functions of Corundum Single Crystals in the Vacuum Ultraviolet Region

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Optical reflection spectra of corundum single crystals grown by HOC and Verneil methods are investigated before and after irradiated by electrons and neutrons using synchrotron radiation polarization (5-30eV).

The peculiarities of defect formation and compositions of surface electron structure and the main tendencies of atomic structure change due to surface destruction in irradiated corundum crystals are considered.

Using the Kramers-Kronig analysis the spectra of optical constants and functions are obtained for nonirradiated-irradiated corundum crystals. The plasmon energy and the energy losses also depend on the orientation of  $\vec{E}$  to  $C_3$ . The effects observed are due to changes in the density of states of the  $2p$ -band electrons.

## I. INTRODUCTION

Aluminum oxide  $\alpha - Al_2O_3$  is of great importance to modern technology. It is using as crystal optical materials, active laser element matrices, radiation stable ceramics, e.t.c.

Recently there has been a considerable interest in studding of the optical properties of corundum and the radiation influence on its electron-energetic structure in ultra violate (UV) and vacuum ultra-violate (VUV) spectra regions [1-8]. Corundum in  $\alpha$ -phase is transparent in VUV spectrum region. It conserves this property under high energy particle radiation.

The phenomenological picture of wave propagation in matter is often used to study the interaction of electromagnetic emission with condensed matter states [9]. The ideal conditions for all phenomenological constants are imposed (proposed). Among them are dielectric  $\varepsilon$  and optical  $n$ ,  $\kappa$  constants. The studding of dielectric and other constants gives the direct information about intrinsic processes in crystals, in particular, corundum. However by some reasons the direct measurement of these constants is difficult task. For example, the very thin matter layers are necessary in order to measure the  $\alpha$ -absorption ( $\approx 10^{-5}cm^{-1}$ ). So, the indirect measurement of optical constants is one of the main problems of the spectroscopy.

There is a sufficiently accurate method to determine the optical constants by means of reflection coefficient of light in wide band region of spectra. One of the main methods applying in the fundamental absorption region is the dielectric constant calculation using Kramers-Kronig relations [10].

In this article the optical constants and optical functions of corundum monocrystal are investigated using the optical reflection spectra and irradiation influence on their energetic structure.

## II. THE METHODS OF INVESTIGATIONS

The relative reflection of crystals was measured by means of synchrotron radiation (SR) of C-60 accelerator. The device used in experiment is based on monochromator constructed by Vodsvoort modification scheme without band, which has the better resolution than 10nm. The resolution of spectra measurement in low energy region was  $\pm 5\%$ , in high energy region it was  $\pm 3\%$  and energy resolution was about  $\pm 0.1eV$ .

The dispersion relations,  $\varphi$  phase,  $n$  and  $k$  constants was calculated using the experimental data of reflection coefficient  $R(E)$  at 300K and Kramers-Kronig dispersion relation. The reflection coefficient is known in the restricted frequency region from  $\omega_{min}$  to  $\omega_{max}$ . Thus the contribution of other regions is essential. The frequencies below  $\omega_{min}$  are in the transparency region of  $\alpha - Al_2O_3$  crystal. At the same time  $\varphi = 0$ , because  $r$  is real and the real values of  $R(E)$  was determined from  $n$  coefficients, which was measured independently using Frenel formulae. The high frequency approximation ( $E > 30eV$ ) is  $R(E) \sim \omega^{-4}$ .

The optical functions  $n$ ,  $k$ ,  $\varepsilon_1$ ,  $\varepsilon_2$ ,  $Im(\varepsilon)^1$ ,  $Im(\varepsilon + 1)^1$ ,  $N_{eff}$  [10-12] were determined from  $\Theta(E)$  in 5 – 30eV region, where  $R(E)$  was measured.

For  $\alpha - Al_2O_3$  the molecule density is  $2.3 \times 10^{22}cm^{-3}$ .

For the  $R(E)$  measurement the  $\alpha - Al_2O_3$  samples, which were grown by means of horizontally oriented crystallization (HOC) and Verneil methods, was used. The optical axes  $C_3$  was parallel to large side of crystal. The samples had a good mirror surface. The crystals were cultivated by  $HCl$  and washed by pure water before the reflection spectra measurements.

The corundum monocrystals were irradiated by electrons of 50MeV energy at 273K and by reactor neutrons of energy 2MeV at 373K.

## III. RESULT DISCUSSION.

Fig.1 and Fig.2 contain the experimental reflection spectra  $R(E)$  of HOC and Verneil crystals. It can be observed from the pictures that a weak maximums (bands) of 5.4eV;

6.3eV; 7eV for Verneil and of 6.1eV for HOC crystals appear in low energy band of spectra  $R(E)$ .

These bands for irradiated crystals we related to  $F^\pm$ ,  $F^{2+}$  and  $F$  centers [13]. The appearance of bands 6–6.5eV and 7–7.4eV is caused by ferrum group ions  $Cr, Mn, Fe, Ti, Ni, Co$ , which have different anisotropy.  $Cr^{3+}$  ion concentration in Verneil samples was  $\sim 10^{17}cm^{-3}$ , in HOC samples was  $\sim 10^{15}cm^{-3}$ .

After radiation the  $R(E)$  spectra maximums, mentioned above, increase and become more precise (the curves). These are the absorption bands of 5.4eV; 6.3eV; 7eV and 8eV, caused by  $F^+$ -center and the band of 6.1eV, caused by  $F$ -center.

The radiation and essentially electron, neutron action changes the reducible center medium stronger than it is expected by the elastic interaction theory.

Note, that the defect formation under the neutron radiation of corundum is much more faster than one under the high speed electron radiation at same doses. It can be seen from the experimental data of  $R(E)$  that the electric field changes intensively on near-surface layers due to defect compound. This affects on point color centers.

In  $R(E)$  spectra of  $\alpha - Al_2O_3$  the intensive long wave peak at 8.9 – 9.1eV appears. The exciton peak depends on SR polarization and orientation of crystal (Fig.1,2). After the radiation the shift of exciton band maximum on  $\pm 0.2eV$  can be observed. The anion  $\Gamma$ -excitons are created, because the high valent band of  $Al_2O_3$  is formed by  $2p$  states of oxide anions [14]. It is known that the ... of exciton bands is the consequence of the selection rule, according to which the during the absorption photon quasimomentum is conserved and translated to exciton.

The large bandwidth and inhomogeneosy of exciton spectra absorption is explained by mixed (hybrid) character of exciton states. It is known that that the hybrid excitons as a "sum" of hyperbolic and parabolic ones are observed at the energetical degeneracy of critical points  $M_0$  and  $M_1$ . Hyperbolic excitons are not dynamically stable and decompose on free electrons and holes. The mixed character of exciton absorption bands can be explained by different bond energies of excitons  $M_0$  and  $M_1$ .

There is a nonelementar wide-band maximum in the reflection spectra of irradiated crystals in the region  $12 - 25\text{eV}$ . Band group in  $R(E)$  of HOC crystals is more precise than in Verneil samples. This fact is connected with the presence of doped ions on the surface of Verneil crystals, which leads to the stoichiometry violation in anion and cation sublattices.

From the pictures, the values of reflection coefficients in radiated samples begin to fall. It proves the radiation influence on crystal surface. Besides the near-surface defects the scattering effect increases too, which also contributes to reflection coefficient.

During the (electron, neutron) radiation of corundum the crystal structure disorder of near-surface increases because of high concentration of vacancies and interband ions. The radiation stimulated desorption process takes place with recovered  $Al$  phase as a result [5,6].

Besides the point vacancy defects the complicated color centers of  $[Al_iF]$  type with various charge states can be formed [14].

Other mechanism of defect formation in near-surface layers is also possible. It is based on dislocation formation model [15,16].

The consequence of corundum surface decoration by  $Al$  ions is the reflection improvement of radiated crystals at energy  $\hbar\nu > 25\text{eV}$ .

The optical constants, calculated from experimental  $R(E)$  changes also (Fig.3-8). The band group is well observed in  $n, \kappa, \varepsilon_1, \varepsilon_2$  spectra also.

It is easy to see from Fig.3,4 that the optical constant  $n$  has thin structure at different orientations between  $\vec{E}$  and  $C_3$ . The high resolution reflection spectra was measured owing to both higher intensity and polarization of SR than in [10,11]. The difference in  $n, \kappa$  spectra on Fig.3 and 4 is conditioned by the value of special energetic state of surface, which affects on radiation irregularity formation in HOC and Verneil samples.

The reflection band group (Fig.1,2) in region  $12 - 25\text{eV}$  is showed precisely in  $n, \kappa, \varepsilon_1, \varepsilon_2$  spectra also (Fig.3-8). According to calculations, the values of  $\varepsilon_1$  and  $\varepsilon_2$  differs for different orientations between  $\vec{E}$  and  $C_3$ . The radiation influence on dielectric constant is essential. Up to energy  $8.2\text{eV}$   $\varepsilon_2$  and  $\kappa$  vanish. It is easy to see from the  $\varepsilon_2$  spectra, that for HOC in

case of  $E \perp C_3$  the energy is  $23\text{eV}$  and in case of  $E \parallel C_3$  it is  $21\text{eV}$ . For Verneil samples the  $\varepsilon_2$  value corresponds to  $23\text{eV}$  and  $20\text{eV}$  and after the electron radiation it becomes  $21\text{eV}$  and  $21.5\text{eV}$ .

The data, reduced from optical constant spectra (Fig.5,8), confirm that the polarization effects are connected with splitting of valence  $2p$ -states of oxygen in low symmetry fields of corundum lattice.

The interband state maximal density change effect probably is connected with the transitions from the highest intensive maximum in density of states of  $2p$ -band. This is due to increase of defect concentration in near-surface layers (especially for neutron radiation).

In order to estimate quantitatively the interband intervals the calculations of interband combined density  $\varepsilon_2 E^2$  (Fig.9,10) and energetic loss functions  $Im(\varepsilon)$  and  $Im(\varepsilon + 1)^{-1}$  (Fig.11,12).

It can be seen from the energetic loss functions of electrons in HOC crystals that in case of  $E \perp C_3$  the largest value of volumetric plasmon  $Im(\varepsilon)^{-1}$  of radiated and irradiated crystal are  $25\text{eV}$  and  $24.5\text{eV}$  correspondingly (the curves 1,3). The maximums of surface plasmon  $Im(\varepsilon + 1)^{-1}$  of radiated and irradiated crystal are  $24\text{eV}$  and  $23.5\text{eV}$  correspondingly (the curves 2,4). In case of  $E \parallel C_3$  for  $Im(\varepsilon)^{-1}$  one has maximums at  $25\text{eV}$ ,  $23.5\text{eV}$  (the curves 1,3) and for  $Im(\varepsilon + 1)^{-1}$  one has maximums at  $23.5\text{eV}$ ,  $23\text{eV}$ .

For Verneil crystals in the energetic loss functions the maximums in case of  $E \perp C_3$  (Fig.12) for  $Im(\varepsilon)^{-1}$  are  $26\text{eV}$  and  $24.5\text{eV}$  (the curves 1,3) and for  $Im(\varepsilon + 1)^{-1}$  its are  $25\text{eV}$  and  $24.5\text{eV}$  (the curves 2,4). In case of  $E \parallel C_3$  (Fig.12) for  $Im(\varepsilon)^{-1}$  one has maximums at  $25.5\text{eV}$ ,  $24.5\text{eV}$  (the curves 1,3) and for  $Im(\varepsilon + 1)^{-1}$  one has maximums at  $25\text{eV}$ ,  $24\text{eV}$ .

Note, that the value  $Im(\varepsilon)^{-1}$  is proportional to the energy loss probability of quasi-free gas electrons in  $\alpha - \text{Al}_2\text{O}_3$ . It describes the maximal fluctuation of plasmon energy, which constitutes  $26\text{eV}$  at nonpolarized light.

Besides the intensive plasma fluctuations in  $Im(\varepsilon)^{-1}$  spectra the weak peaks are observed in the regions  $9.8$ ,  $13$ ,  $14.5$ ,  $15.5$ ,  $20$ ,  $21.5\text{eV}$ . Its are the consequences of energy loss of near-

surface and volumetric  $Al$  plasmons of nonirradiated and irradiated corundum crystals.

The characteristic loss spectra shows that  $Al$  plasmon peak location depends drastically on both type and dose of radiation action and leads to shift on  $0.2 - 0.5eV$ .

The effective electron number  $N_{eff}$  is calculated from the sum rule, which express the fact that the sum of all oscillator forces equals to electron number.

In the plasmon energy region  $N_{eff}$  reaches 14 electrons per  $Al_2O_3$  molecule, which has 18 valence  $2p$ -electrons. The fact that its are not saturated means the oscillator are not entirely strengthless. Hence, the absorption, which contributes to the sum rule, must be below and higher  $30eV$  (Fig.13).

Using Arand curves for HOC and Verneil crystals the resonance energies of partial oscillators have been precisely determined. Every semicircle on Fig.14-17 corresponds to one oscillator, i.e. one interband transition.

To calculate the resonance energies one first have extrapolated every semicircle and then have found the values  $O_i$  using maximum  $\varepsilon_\alpha(E)$ .

In tables 1,2 the identifications of interband transitions for HOC and Verneil crystals are presented for two polarizations ( $\vec{E} \perp C_3, \vec{E} || C_3$ ) taking into account the electron energetic structure of corundum.

Thus, our identification of interband transition for corundum crystals shows two effects of polarization and radiation. The first one is connected with splitting of the 2d valence electrons of oxygen in the low symmetry crystal field of corundum. The second one is related to transition in the upper intensive maximum of the 2p band state density after increasing the radiation defects in near-surface layers.

TABLE I. HOC nonirradiated and irradiated by neutrons

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$O_1^\perp - O_1^\parallel$	9.20 – 9.0 eV	$O_{1,rad}^\perp - O_{1,rad}^\parallel$	8.60 – 9.0 eV
$O_2^\perp - O_2^\parallel$	11.60 – 10.0 eV	$O_{2,rad}^\perp - O_{2,rad}^\parallel$	9.0 – 9.4 eV
$O_3^\perp - O_3^\parallel$	12.60 – 12.80 eV	$O_{3,rad}^\perp - O_{3,rad}^\parallel$	12.80 – 12.60 eV
$O_4^\perp - O_4^\parallel$	18.40 – 18.60 eV	$O_{4,rad}^\perp - O_{4,rad}^\parallel$	18.40 – 19.20 eV
$O_5^\perp - O_5^\parallel$	21.80 – 21.0 eV	$O_{5,rad}^\perp - O_{5,rad}^\parallel$	21.40 – 21.20 eV
$O_6^\perp - O_6^\parallel$	22.0 – 21.40 eV	$O_{6,rad}^\perp - O_{6,rad}^\parallel$	22.80 – 22.20 eV
$O_7^\perp - O_7^\parallel$	23.0 – 23.20 eV	$O_{7,rad}^\perp - O_{7,rad}^\parallel$	26.60 – 24.0 eV
$O_8^\perp - O_8^\parallel$	24.60 – 24.40 eV	$O_{8,rad}^\perp - O_{8,rad}^\parallel$	28.40 – 25.60 eV

1-3  $[O^\perp - O^\parallel] - [8.60\text{--}13.0] \text{ eV} - 2p[O^2]$ ;

4-5  $[O^\perp - O^\parallel] - [18.0\text{--}20.5] \text{ eV} - 3s[Al^{3+}] - 2s[O^{2-}]$ ;

5-8  $[O^\perp - O^\parallel] - [21.0\text{--}28.5] \text{ eV} - 3sp^2[Al^{3+}] - 2s[O^{2-}]$ .

TABLE II. Verneyl: nonirradiated and irradiated by electrons

$O_1^\perp - O_1^\parallel$	9.40 – 9.20 eV	$O_{1,rad}^\perp - O_{1,rad}^\parallel$	9.60 – 9.20 eV
$O_2^\perp - O_2^\parallel$	11.0 – 11.30 eV	$O_{2,rad}^\perp - O_{2,rad}^\parallel$	11.20 – 11.60 eV
$O_3^\perp - O_3^\parallel$	12.20 – 12.0 eV	$O_{3,rad}^\perp - O_{3,rad}^\parallel$	12.0 – 11.80 eV
$O_4^\perp - O_4^\parallel$	16.80 – 17.0 eV	$O_{4,rad}^\perp - O_{4,rad}^\parallel$	19.40 – 19.20 eV
$O_5^\perp - O_5^\parallel$	19.80 – 18.80 eV	$O_{5,rad}^\perp - O_{5,rad}^\parallel$	20.20 – 20.0 eV
$O_6^\perp - O_6^\parallel$	21.0 – 19.20 eV	$O_{6,rad}^\perp - O_{6,rad}^\parallel$	21.0 – 21.20 eV
$O_7^\perp - O_7^\parallel$	21.40 – 20.40 eV	$O_{7,rad}^\perp - O_{7,rad}^\parallel$	22.80 – 21.60 eV
$O_8^\perp - O_8^\parallel$	22.60 – 22.20 eV	$O_{8,rad}^\perp - O_{8,rad}^\parallel$	24.80 – 23.80 eV
$O_9^\perp - O_9^\parallel$	23.40 – 24.0 eV	$O_{9,rad}^\perp - O_{9,rad}^\parallel$	– 24.80 eV
$O_{10}^\perp - O_{10}^\parallel$	26.0 – 25.80 eV	$O_{10,rad}^\perp - O_{10,rad}^\parallel$	—



1-3  $[O^\perp - O^\parallel] - [9.0-12.5] \text{ eV} - 2p[O^2]$ ;  
 4-5  $[O^\perp - O^\parallel] - [16.5-20.5] \text{ eV} - 3s[Al^{3+}] - 2s[O^{2-}]$ ;  
 6-10  $[O^\perp - O^\parallel] - [21.0-26.0] \text{ eV} - 3sp^2[Al^{3+}] - 2s[O^{2-}]$ .

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#### IV. FIGURE CAPTIONS

FIG. 1. Reflection spectra vs. Polarization of SR with respect to the optical axis of corundum crystals (HOC)  $C_3$ . (1)-nonirradiated; (2)-irradiated dose  $10^{17}n/cm^2$

FIG. 2. Reflection spectra vs. Polarization of SR with respect to the optical axis of corundum crystals (Verneil)  $C_3$ . (1)-nonirradiated; (2)-irradiated dose  $3 \cdot 10^{17}el/cm^2$

FIG. 3. Optical constants of corundum single crystals, (HOC): 1(**n**), 3(**k**)-nonirradiated; 2(**n**), 4(**k**)-irradiated dose  $10^{17}n/cm^2$ .

FIG. 4. Optical constants of corundum single crystals, (Verneil): 1(**n**), 3(**k**)-nonirradiated; 2(**n**), 4(**k**)-irradiated dose  $3 \cdot 10^{17}el/cm^2$ .

FIG. 5. Dielectric constants of corundum single crystals (HOC): 1( $\epsilon_1$ ), 3( $\epsilon_1$ )-nonirradiated; 2( $\epsilon_1$ ) (1), 4( $\epsilon_1$ ) (2)-irradiated dose  $10^{17}n/cm^2$ .

FIG. 6. Dielectric constants of corundum single crystals (HOC): 1( $\epsilon_1$ ), 3( $\epsilon_2$ )-nonirradiated; 2( $\epsilon_1$ ), 4( $\epsilon_2$ ) irradiated dose  $10^{17}n/cm^2$ .

FIG. 7. Dielectric constants of corundum single crystals (Verneil): 1( $\epsilon_1$ ), 3( $\epsilon_2$ )-nonirradiated; 2( $\epsilon_1$ ), 4( $\epsilon_2$ )-irradiated dose  $3 \cdot 10^{17}el/cm^2$ .

FIG. 8. Dielectric constants of corundum single crystals (Verneil): 1( $\epsilon_1$ ), 3( $\epsilon_2$ )-nonirradiated; 2( $\epsilon_1$ ), 4( $\epsilon_2$ )-irradiated dose  $3 \cdot 10^{17}el/cm^2$ .

FIG. 9. Interband combined density ( $\epsilon_2 E^2$ ) (HOC): 1,3-nonirradiated; 2,4-irradiated dose  $10^{17}n/cm^2$ .

FIG. 10. Interband combined density ( $\epsilon_2 E^2$ ) (Verneil): 1,3-nonirradiated; 2,4-irradiated dose  $3 \cdot 10^{17}el/cm^2$ .

FIG. 11. Energetic loss functions of corundum single crystals (HOC): 1,2-nonirradiated; 3,4-irradiated dose  $10^{17}n/cm^2$ .

FIG. 12. Energetic loss functions of corundum single crystals (Verneil): 1,2-nonirradiated; 3,4-irradiated dose  $10^{17}n/cm^2$ .

FIG. 13. Effective electron number of  $\alpha - Al_2O_3$  single crystals (HOC, Verneil): 1,2-nonirradiated:  $\vec{E} \parallel C_3$  and  $\vec{E} \perp C_3$ ; 3,4-irradiated:  $E \parallel C_3$  and  $\vec{E} \perp C_3$ .

FIG. 14. Argand diagram for corundum crystals (HOC,  $\vec{E} \perp C_3$ )  $a$ -nonirradiated,  $b$ -irradiated  $10^{17}n/cm^2$ .

FIG. 15. Argand diagram for corundum crystals (HOC,  $\vec{E} \parallel C_3$ )  $a$ -nonirradiated,  $b$ -irradiated  $10^{17}n/cm^2$ .

FIG. 16. Argand diagram for corundum crystals (Verneil,  $\vec{E} \perp C_3$ )  $a$ -nonirradiated,  $b$ -irradiated  $3 \cdot 10^{17}el/cm^2$ .

FIG. 17. Argand diagram for corundum crystals (Verneil,  $\vec{E} \parallel C_3$ )  $a$ -nonirradiated,  $b$ -irradiated  $3 \cdot 10^{17}el/cm^2$ .

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